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# **EXHIBIT 3**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

David R. Battiste

Serial No.: 09/705,316

Filed: November 3, 2000

For: IMPROVED MONITORING AND  
CONTROL OF PROCESSES FOR  
MAKING 1-HEXENE


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Group Art Unit: 1764

Examiner: Dang, Thuan D.

Atty. Docket: CPCM:0008/FLE  
33938US

Mail Stop AF  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-145031

CERTIFICATE OF MAILING 37 C.F.R. 1.8	
I hereby certify that this correspondence is being deposited with the U.S. Postal Service, with sufficient postage, as First Class Mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on the date below	
March 24, 2004	
Date	Helen Tinsley

Sir:

**DECLARATION UNDER 37 C.F.R. § 1.131**

I, David R. Battiste, hereby declare as follows:

1. My residence address and citizenship are set forth below, under my signature.
2. I am the sole inventor of the subject matter set forth in the above-referenced application.
3. I conceived of the subject matter disclosed and claimed in the above-referenced application at least as early as July 19, 1999. This conception is evidenced by four (4) pages from my laboratory notebook, which record the calibration results of a Low Resolution Raman

Spectrometer in preparation of an actual reduction to practice of the claimed subject matter. A redacted copy of the four (4) pages from my laboratory notebook is attached hereto as Exhibit A.

4. I actually reduced to practice the subject matter disclosed and claimed in the above-referenced application at least as early as August 11, 1999. My actual reduction to practice is evidenced by three (3) pages from my laboratory notebook, which record the results of a successful test of an implementation of the claimed subject matter. A redacted copy of the three (3) pages from my laboratory notebook is attached hereto as Exhibit B.

5. All acts related to my conception and actual reduction to practice of the invention took place in the United States of America.

6. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements are made with the knowledge that willful false statements, and the like, are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

Dated: 2/27/04

By: David R. Battiste  
David R. Battiste

Declarant's Full Name: David R. Battiste

Country of Citizenship: U.S.A.

Residence Address: 307 Stoneridge Court,  
Bartlesville, Oklahoma 74006

# EXHIBIT A

## Low Resolution Raman Project

BBA Annotations

Spectrum Time (min)	File	Comment	Time
10	CyC688	Pure $\text{C}_2$ ; 97.33%/2.67% Dodecane	
10	CyC688F	" Internal Standard IS=2.67%	7:10A
60	CyC660	"	
60	CyNC2A21	$\text{C}_2 = 1.53\%$ / $\text{C}_2 = 96.02\%$ / IS=2.45%	8:01
60	CyNC2A22	" " " "	
60	High C2 Level	$\text{C}_2 = 20.44\%$ / $\text{C}_2 = 78.20\%$ / IS=1.19%	
20	C2at20	Pure $\text{C}_2$ , 400 psig, 450g/hr flow	
20	C2at20A		
20	C2at20B		
60	C2at60		8:30A
60	C2at60A		Ch
60	C2at60B		
60	C2at60C		pres
30	CYC2a20	Cyclohexane + $\text{C}_2$ ; $\text{C}_2$ at .3 gal/hr	11:3
20	CYC2a20A	$\text{C}_2$ at 450g/hr	
20	CYC2a20B	450 psig back pressure	
60	CYC2a60		
60	CYC2a60A		
36 hrs	60	CYC2a60B - GC sample $\text{C}_2 = 19.21\%$ / $\text{C}_2 = 79.62\%$	
	20	CYC2a20C IS=1.05%	
	30	CYC2a30	132A
16 hrs	30	C6C2a30 Changed $\text{C}_2$ level	
	30	C6C2a30A GC Sample; $\text{C}_2 = 11.58\%$ / $\text{C}_2 = 87.28\%$	
	20	C6C2a20 IS=1.08%	
	20	C6C2a20A	
	60	C6C2a60	
	60	C6C2a60A	
12 hrs	30	CYC2b30 GC Sample; $\text{C}_2 = 8.12\%$ / $\text{C}_2 = 90.62\%$	
	30	CYC2b30A IS=1.21%	
	30	CYC2b30B (C)	
	60	CYC2b60	
	60	CYC2b60A	
18	30	CYC6d30 Shut off $\text{C}_2$ at 1418 to get $\text{C}_2$	
	20	CYC6d20	
	20	CYC6d20A	
	60	CYC6d60	
	60	CYC6d60A	
	60	CYC6d60B - GC sample $\text{C}_2 = 0.40\%$	
		$\text{C}_2 = 97.65\%$	
		IS=1.95	
		IS = Internal Standard	
Spectrum Time (min)	File	Comment	Time
30	CYC2c30	GC $\text{C}_2 = 5.53\%$	
30	CYC2c30A	$\text{C}_2 = 93.11\%$	
20	CYC2c20	IS=1.34%	
60	CYC2c60		14C
60	CYC2c60A		14C
20	CYHX20	GC	
20	CYHX20A	$\text{C}_2 = 32.65\%$	
30	CYHX30	$\text{C}_2 = 46.43\%$	
30	CYHX30A	IS=0.92%	
60	CYHX60		141
			St

## Measurement of LRR Spectrum of Cyclohexane/Hexene Blends with Ethylene Added

Time	Spectral Time	File	Comment
7:10 AM			(rate = 3 gal/hr) Start the 25% hexene in cyclohexane solution + add ethylene to it at a rate of 112 g/hr through hexene reactor in 88°F (G. Cowan)
8:01 AM	30	Blend001	- GC sample taken. Actual concentration measured by GC $C_2^- = 5.44\%$ / $C_6^- = 21.35\%$ / $C_7C_6 = 72.22\%$ / $IS = 0.89$
	30	Blend002	Second spectrum, same condition. $Decom = 0.01$
	60	Blend003	First 60 sec spectrum
	60	Blend004	Second " " "
8:30 AM			Changed to the 225 g/hr rate of $C_2^-$ addition from the starting 112 g/hr rate. 450 psig back pressure. Room temperature. Had to go to phone conference. Returned at 11:15. Reactor purged.
	30	Blend005	Taken before GC sample run.
11:31 AM	30	Blend005	- GC sample: $C_2^- = 16.91\%$ / $C_6^- = 19.53\%$ / $C_7C_6 = 62.50\%$ / $IS = 0.99\%$
	60	Blend006	60 sec. spectrum
	60	Blend007	" " "
1320 hrs	30	Blend008	- GC sample at 450 g/hr rate of $C_2^-$ . $C_2^- = 24.54\%$ / $C_6^- = 16.85\%$ / $C_7C_6 = 56.99\%$ / $IS = 1.25$
	30	Blend009	
	60	Blend010	
		011	
		012	
1400			Switched to nominal 15% $C_6^-$ blend in $C_7C_6$ , run with no $C_2^-$ added
1403 hrs	60	Blend013	- GC sample at 0 rate of $C_2^-$ addition.
	60	014	$C_2^- = 0.01\%$ / $C_6^- = 14.69\%$ / $C_7C_6 = 83.97\%$ / $IS = 1.33\%$
	30	015	
	30	016	
1410 hrs			Started adding $C_2^-$ at 112 g/hr rate.

ument

2.8  
5.53%

13.11%

1.34%

2.65%

6.43%

2.92%



Continue page 19

Time	Spectral Time	File	Comment
1452	30	Blend017	GC Sample run
	30	Blend018	$C_2 = 2.98\% / C_6 = 14.53\% / \text{IS} = 81.18\% / \text{IS} = 1.27\%$
	60	Blend019	" " "
	60	Blend020	" " "
1505 hrs	Changed to	225 g/hr $C_2$ added level.	
	60	Blend021	$C_2 = 13.69\% / C_6 = 12.20\% / \text{IS} = 73.13\% / \text{IS} = 0.89\%$
	30	022	
	30	023	
1558 hrs	Changed to	450 g/hr rate $C_2$ added. PSIG = 400.	
1643 hrs	30	Blend024	GC sample Run!
	30	Blend025	$C_2 = 20.06\% / C_6 = 10.91\% / \text{IS} = 67.83\% / \text{I.S.} = 1.03\%$
	60	Blend026	
	60	Blend027	
1350	30	Blend028	30 sec. stable 112 g/hr $C_2$ into 15% Stock Soln of $C_2$ in $C_6$ This is with dark current used for Hydrogen spectra.
	30	Blend029	Re established dark current + took spectrum with GC analyze
	30	Blend030	- GC shot: 7.38% $C_2$ / 12.77% $C_6$ / 77.50% $C_6$
	30	Blend031	
	30	Blend032	Changed to
	Changed to	450 g/hr $C_2$ rate at 1412 hrs.	
	30	Blend033	- GC sample taken: 20.06% $C_2$
	30	Blend034	10.98% $C_6$
	60	Blend035	67.70% $C_6$
	60	Blend036	
Hydrogen spectrum taken of standard 1A cylinder Hydrogen at 100 psig at Raman probe.			
Files: Hydrogen1 / H2 at 60.1 / H2 at 60.22 / H2 at 30.1 / H2 at 30.12 /			
60 sec 60 sec 50 sec 80 sec			

# Continued LRR Spectra Collection: 88F Set 46

(min)	Spectral File	Comment
1045	30 IC4001	Isobutane + Hexene. GC sample: IC4 = 94.19% / Butene = 0.58% / another butene = 0.08% and $C_6^+ = 5.13\% / C_2^+ = 0$
	30 IC4002	
	60 IC4003	
	60 IC4004	
11:15	60 IC4005	changed to feed 30g $C_2^+$ / hr actually ~ 76g / 2 hr pumped - GC Sample at 1330 hrs. 76g $C_2^+$ in 2 hr
11:30	60 IC4006	IC4 = 86.15% / Butene = 0.43 + 0.09% / $C_2^+ = 4.98\% / C_6^+ = 5.06\% / C_7^+ C_8^+ = 1.20\%$
1340	Change $C_2^+$ feed rate to 65 g/hr at 1340 hrs.	
1442	60 IC4007	
	60 IC4008	- GC sample taken: IC4 = 84.65% / Butene = 0.41 + 0.08% / $C_2^+ = 7.27\%$
	60 IC4009	$C_6^+ = 4.80\% / C_7^+ C_8^+ = 2.67\%$
1445	Increased $C_2^+$ to 112 g/hr feed rate	
1530	60 IC4010	- GC Sample at 1530 hrs: IC4 = 83.49% / Butene = 0.46 + 0.07% / $C_2^+ = 11.04$
	60 IC4011	$C_6^+ = 4.77\% / C_7^+ C_8^+ = 0.13\%$
1535	Changed to straight isobutane with no hexene and no ethylene.	
	60 IC4012	- GC Sample at 1556 hrs.
	60 IC4013	
	60 IC4014	Still isobutane
1608	Change to mixed decenes. Blow out line @ $N_2$ then put in decenes	
	Nitrogen spectrum saved	
	60 Nitrogen	
	60 Decenes	- Mixed decenes to determine whether or not there is a
	60 DecenesA	possibility to detect these compounds.
	30 Run001	First sample $C_7^+ C_8^+ = 85.98, C_2^+ = 2.27, C_6^+ = 13.54$
	30 Run002	GC sample $= 85.85, = 2.57 = 13.32$

Saved spectrum of IC4 None when we saw no peaks for straight isobutane.

probe.

# EXHIBIT B

# Second Reaction Monitoring $C_2^-$ to $C_6^-$ with R-2000

Purpose: To start with low catalyst concentration to give R-2000 chance to detect high ethylene level + low heptene level. We want to use these data to improve quantitative model.

Time	Run #	Comment	Conte
7:55A	ARXN000	Hot cyclohexane wash of reactor, 150°C, to flush poisons after 3 cc TEA treatment	9:55A
8:05A	ARXN001	Second spectrum during hot cyclohexane wash; contains $C_2$ , $C_6$ + dodecane	9:58A
8:10A	ARXN002	Third hot wash spectrum just before putting catalyst in reactor at 8:11 AM	10:17
8:21	ARXN003	Spectrum with catalyst being pumped into reactor with cyclohexane + dodecane	10:58
8:27	ARXN004	Still only cyclohexane + catalyst	11:45
8:37	ARXN005	Still only cyclohexane + catalyst	12:23
8:42	ARXN006	Last sample before $Cr^-$ and $H_2$ added	13:00
8:50	ARXN007		13:32
8:55	ARXN008		14:12
9:00	ARXN009		14:5
9:05	ARXN010		15:4
9:10	ARXN011		16:2
9:15	ARXN012		8/12 PM ~ 8:20
9:20	ARXN013		8:10
9:35	ARXN014	- Reset dark current	8:07 8:14
9:37	ARXN015	- Took GC sample { $C_2^-$ / $C_6^-$ / $C_7C_8$ / 2 ethyl heptene / internal dodecane 09/11/91 10:18:01.0 { 32.44 / 0.099 / 62.11 / 4.60 / 0.586	8:4
9:39	ARXN016	- Next spectrum for duplicate run.	9:13
9:40	ARXN017		10:15

ARX

Set up Time Acquisition: Toggle Laser On/Off [R]; Laser Warmup Time 40 sec; Integration 30 sec;  
Store Dark Before En Acq. [R] Delay before 1<sup>st</sup> Acq. = 1 sec; Delay between  
Acquisitions = 180; Number of Acq. = 520; File C:\Program Files\R2000\ARX\A.exp

conversion of  
Reaction at 88F  $C_2^-$  to  $C_6^-$

Test of Model		Predicted			Analyzed by GC				Dodecanal IS	
Time	Spl	CyC <sub>6</sub>	C <sub>2</sub> <sup>-</sup>	C <sub>6</sub> <sup>-</sup>	CyC <sub>6</sub>	C <sub>2</sub> <sup>-</sup>	C <sub>6</sub> <sup>-</sup>	C <sub>10</sub> <sup>-</sup>		
~10:55 AM	Run001									
~10:56 AM	Run002	85.5	2.57	13.54	83.71	3.99	10.22	0.774	1.29	Head NIR cell in line
11:22	Run003	81.60	3.43	17.30						
11:31	Run004	79.93	4.40	17.48						
11:40	Removed 9 ml sample cell of near IR from line to keep from having lag time from GC to Raman probe. Sample pressure 450 psig.									
11:45	Run005	82.03	2.61	19.78						
11:52	Run006	80.52	3.44	19.89						
11:57	Run007	80.59	3.55	19.63	* Total GC 79.006	4.53	13.65	1.57	1.11	
12:06	Run008	80.60	3.62	19.36						
12:15	Run009	80.49	4.25	18.48						
12:30	Run010	80.24	4.28	17.47						
12:45	Run011	81.77	3.28	17.14						
13:00	Run012	78.67	4.53	18.22	* Total GC 77.824	5.15	14.707	1.320	1.020	
13:15	Run013	77.18	4.80	17.98						
13:30	Run014	77.75	5.18	17.93						
13:45	Run015	78.11	5.11	17.26						
14:00	Run016	77.81	5.08	18.17	* Total GC 77.35	5.31	14.95	1.25	1.06	
14:15	Run017	77.95	4.85	18.08						
14:30	Run018	77.83	4.75	18.41						
14:45	Run019	79.02	4.47	17.27						
15:00	Run020	79.11	4.67	17.30	* Total GC 78.87	5.28	13.52	1.19	1.07	
15:15	Run021	78.59	5.16	17.36						
15:30	Run022	77.50	5.07	18.21						
15:45	023									
16:00	024				* Total GC 76.88	5.86	14.71	1.20	1.03	

Continued: Set up automatic agitation every 150 sec.

	Time	Scan#	File	GC File 8/11/99	C <sub>2</sub>	C <sub>6</sub>	C <sub>10</sub>	Int. Std Decadecane	Decadecane
M	9:55A	0	ARXA00001 <del>ARXA00001</del>						
nlc	9:58A	1	00002						
del.	10:17	5	00006	002B0201.D	12.968	3.449	66.242	9.17(?)	1.355 0.050
	10:56	17	00017	003B0301.D	7.617	15.475	69.551	4.81	1.013 .751
	11:45	30	00030	004B0401.D	7.910	17.387	70.024	2.078	.974 .105
thrust	12:23	41	00041	005B0501.D	6.948	18.335	69.645	2.248	.961 .124
can	13:00	51	00051	006B0601.D	6.112	18.991	68.423	2.499	1.049 1.804
8:11AM	13:32	61	00061	007B0701.D	6.081	18.290	71.186	1.181	1.009 1.945
reladecane	14:12	71	00071	008B0801.D	5.599	19.208	70.775	1.046	1.027 2.025
	14:50	82	00082	009B0901.D	5.612	18.966	71.122	.477	1.021 1.979
	15:42	96	00096	010B1001.D	6.386	20.444	68.638	1.049	.946 2.177
	16:21	107	00107	011B1101.D	6.319	21.175	67.722	1.129	.928 2.309
	~8:00	auto start	ARXB						
	8:07	2	00002	91K					
	8:49	12	00002	013B1301.D	8.1654	23.879	64.942	.016	.857 1.314
	8:49	14	00014	014B1401.D	8.201	3.8053	48.501	.016	.604 3.984
	9:39	28	00028	015B1501.D	8.533	50.745	29.238	-0-	.404 9.789
	10:21	39	00039	016B1601.D	6.466	51.846	29.893	-0-	.398 10.019
	11:00	50	00050	Bled down to clean filter.					
	11:06	52	00052	017B1701.D	5.677	52.733	25.433	-0-	.365 13.998
decane	11:50	64	00064	018B1801.D	5.609	52.182	26.405	-0-	.372 13.397

Started scans, file name ARXB\*.exp: 7:18AM  
Gfmd started rxn

show scans  
begin between  
as  
ARXA.00001